TRANSLATION

(19) KOREAN INDUSTRIAL PROPERTY OFFICE (KR)

(12) OFFICIAL GAZETTE OF UNEXAMINED PATENTS

(11) PATENT NO. 1998-049300 (A)

(51)	Int. Cl.[4]:	D 01 F 6/62
(21)	Filing No.:	1996-067992
(22)	Filing Date:	December 19, 1996
(43)	Disclosure Date:	September 15, 1998

(54) MANUFACTURING METHOD OF DRAWN POLYTRIMETHYLENE TEREPHTHALATE FIBER

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The present invention relates to the manufacturing method of polytrimethylene terephthalate (PTT) drawn fiber, wherein greater than 85% of the main chain of the polytrimethylene terephthalate is made of 1,3-propanediol, the intrinsic viscosity of the polytrimethylene terephthalate is 0.75-1.1, and such polytrimethylene terephthalate is spun at a spinning rate of 2500-5500 m/min to obtain POY, followed by drawing. This method allows reliable manufacturing of drawn fibers with excellent winding property from polytrimethylene terephthalate.

Title of the invention

Manufacturing method of drawn polytrimethylene terephthalate fiber

Detailed explanation of the invention

The present invention relates to a manufacturing method of polytrimethylene terephthalate (PTT) drawn fiber. In more detail, the present invention concerns a reliable manufacturing method of drawn fiber with good winding property from polytrimethylene terephthalate.

Recently, polytrimethylene terephthalate resin, which is one type of polyester, has become commercialized. Because this resin has excellent flexibility and heat resistance compared to existing polyesters, its application to fibers has been investigated. Particularly, when the fiber is drawn, superior characteristics of PTT can be displayed.

However, the actual drawing temperature for PTT cannot be raised higher than 100°C because of poor heat resistance. As a result, the winding property becomes too poor to display its original flexibility. In addition, it breaks easily during the drawing process, giving a poor appearance. Therefore, many problems have been posed for commercial production.

The present invention is aimed to provide a manufacturing method of drawn polytrimethylene terephthalate fiber without the aforementioned problems.

During the research to achieve the aforementioned goal, the inventors of the present invention found that the strength and heat resistance of PTT fiber is inferior to the existing polyester, but the strength and heat resistance of PTT could be improved by increasing the intrinsic viscosity (IV) of the resin and at higher spinning speed during POY fiber manufacturing. Such POY fiber can be drawn at a temperature higher than 100°C to give a better winding property.

Therefore, the present invention provides a manufacturing method of drawn polytrimethylene terephthalate fiber, which includes spinning of polytrimethylene terephthalate at a rate of 2500-5500 m/min to obtain the POY fiber, followed by drawing, wherein greater than

85% of the main chain of polytrimethylene terephthalate is 1,3-propanediol and the intrinsic viscosity of polytrimethylene terephthalate is 0.75-1.1.

The present invention is described below in more detail.

Polytrimethylene terephthalate employed in the present manufacturing method has an intrinsic viscosity of 0.75-1.1 and contains 1,3-propanediol, of which greater than 80% of the main chain consists. If the intrinsic viscosity is less than 0.75, the molecular weight is low and thereby the drawing process is poor. If the intrinsic viscosity of polytrimethylene terephthalate exceeds 1.1, the manufacturing time, especially polymerization reaction time, becomes longer, which drives up the manufacturing cost.

The ideal spinning rate for manufacturing POY fiber is 2500-5500 m/min. Within the range of spinning rate, the strength of the resulting fiber is greater than 2.2 g/d, and the heat resistance is improved because the maximum thermal stress temperature becomes greater than 45°C. If the spinning rate is less than 2500 m/min, the strength of the resulting fiber is inadequate and the heat resistance is insufficient, which tends to cause fiber breakage and shag formation. In addition, if the spinning rate exceeds 5500 m/min, it does not further improve the strength of the fiber but rather it causes a poor spinning process.

The POY fiber is manufactured in such a way can be drawn at a temperature higher than 100°C without fiber breakage or shag formation. Such drawn fiber exhibits excellent winding property, and stretches more than 30% during winding. It is ideal to adjust the drawing temperature below 200°C if possible. If the fiber is drawn at a temperature higher than 200°C, the fiber may melt.

The innovation and other advantages of the present invention as described above will become more obvious from the following experimental examples. Of course, the following experimental examples are presented to help the reader to comprehend the present invention, but the present invention is not limited to these experimental examples. The properties described in the following experimental examples and comparative examples were measured using the following method.

• Elongation at winding: After the drawn fiber sample is wound at a warp reel, one end of the fiber is attached to the scale plate [illegible], an ultraweight (2/1000 g) and heavyweight (1/10 g) are loaded, and then the length (L_0) of the fiber is measured after 1 min. After the heavyweight is removed, the sample is treated in a boiling water for 20 min and removed. Then, the ultraweight is removed and the sample is dried at room temperature for 24 h. Subsequently, the ultraweight and the heavyweight are loaded again and the length (L_1) of the sample is measured after 1 min. Then, the heavyweight is immediately removed. The length (L_2) of the sample is measured 1 min after the removal of the heavyweight. The winding stretching rate is calculated using the lengths measured above and the following equation.

Elongation at winding = $((L_1-L_2)/L_0) \times 100$

• Intrinsic viscosity (IV): Measured by an Ubbelohde viscometer in a constant temperature bath at 30°C after dissolving the compound in 120°C chlorophenol for 1% concentration,

• Thermal stress: Thermal stress was measured under 10 g of the ultraweight while increasing the temperature at a rate of 300/min [sic] using the thermal stress test device made by Gabone [transliteration] Company.

Experimental Example 1

PTT resin with intrinsic viscosity of 0.76 was spun using a conventional spinneret after drying it in a vacuum oven at 150°C for 6 hours. POY fiber of 115 denier 36 filament was manufactured using 0.3 mm nozzle at the spinning temperature of 265°C and the spinning rate of 3000 m/min. The POY fiber produced in this way was drawn at a rate of 400 m/min at 150°C using a disc time [transliteration] drawing machine. The property and process variables employed in these experimental examples are listed in Table 1 below.

Experimental Example 2

The same procedure as in Experimental Example 1 was repeated, except that the intrinsic viscosity of the PTT resin employed was 1.0, the spinning temperature was 270°C, and the drawing temperature was 160°C.

Experimental Example 3

The same procedure as in Experimental Example 1 was repeated, except that the intrinsic viscosity of the PTT resin employed was 0.76, the spinning rate was 5500 m/min, and the drawing temperature was 160°C.

Experimental Example 4

The same procedure as in Experimental Example 1 was repeated, except that the intrinsic viscosity of the PTT resin employed was 1.0, the spinning rate was 5500 m/min, and the drawing temperature was 150°C.

Comparative Example 1

The same procedure as in Experimental Example 1 was repeated, except that the intrinsic viscosity of the PTT resin employed was 0.65.

Comparative Example 2

The same procedure as in Experimental Example 1 was repeated, except that the intrinsic viscosity of the PTT resin employed was 0.65 and the drawing temperature was 120°C.

Comparative Example 3

The same procedure as in Experimental Example 1 was repeated, except that the intrinsic viscosity of the PTT resin employed was 0.76, and the spinning rate was 2000 m/min.

Comparative Example 4

The same procedure as in Comparative Example 3 was repeated, except that the drawing temperature was 120°C.

Table 1

Classification	Intrinsic viscosity	Spinning rate (m/min)	Strength of POY fiber (g/d)	Maximum temperature of POY fiber thermal stress (°C)	Drawing temperature (°C)	Drawing process	Shag formation	Elongation at winding (%)
Experimental Example 1	0.76	3,000	2.0	53	150	Good	☑	35
Experimental Example 2	1.00	3,000	2.5	53	160	Good	☑	38
Experimental Example 3	0.76	3,500	2.0	61	150	Good	☑	34
Experimental Example 4	1.00	3,500	2.5	61	150	Good	☑	34
Comparative Example 1	0.65	3,000	1.0	49	150	Poor	X	28
Comparative Example 2	0.65	3,000	1.6	49	120	Good	☑	12
Comparative Example 3	0.76	2,000	1.6	41	150	Poor	X	31
Comparative Example 4	0.76	2,000	1.6	41	120	Good	☑	12

O --- No or little formation of shag

X --- Severe formation of shag

O --- No or little formation of shag
X --- Severe formation of shag

As shown in the above Table 1, the drawing result was superior at the drawing temperature higher than 100°C without fiber breakage or shag formation and the drawn fiber showed excellent winding flexibility, greater than 30% of elongation at winding in Experimental Examples 1-4 where the intrinsic viscosity of polytrimethyleneterephthalate of the polyethylene terephthalate was 0.75-1.1, and such polytrimethylene terephthlate was spun at a spinning rate of 2500-5500 m/min to obtain POY fiber, in comparison to Comparative Examples 1-4 where the variables employed were out of the aforementioned range.

Claims

1. A manufacturing method of polytrimethylene terephthalate drawn fiber, which includes spinning of polytrimethylene terephthalate at a spinning rate of 2500-5500 m/min to obtain POY fiber, followed by drawing, wherein the main chain of polytrimethylene terephthalate consists of greater than 85% 1,3-propanediol and the intrinsic viscosity of polytrimethylene terephthalate was 0.75-1.1.

2. A manufacturing method of polytrimethylene terephthalate drawn fiber as claimed in Claim 1, wherein the drawing temperature is 100-200°C.

3. A manufacturing method of polytrimethylene terephthalate drawn fiber as claimed in Claim 1, wherein the strength of the aforementioned POY fiber is higher than 2.2 g/d and the maximum thermal stress temperature is greater than 45°C.

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RALPH MCELROY TRANSLATION COMPANY
MARCH 10, 2000